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(71) Applicant: **ROHM AND HAAS COMPANY**

**Philadelphia, Pennsylvania 19106-2399 (US)**

(72) Inventors:

- **Speece, David Gerald, Jr.**  
**Reading, PA 19606 (US)**

- **Eisenhart, Eric Karl**

**Ambler, PA 19002 (US)**

- **Bowe, Michael Damian**

**Newtown, PA 18940 (US)**

- **Weir, William David**

**Levittown, PA 19056 (US)**

- **Woltersberger, Martha Alice Harbaugh**

**Perkasie, PA 18944-4127 (US)**

(74) Representative: **Buckley, Guy Julian et al**

**ROHM AND HAAS (UK) LTD.**

**European Operations Patent Department**

**Lennig House**

**2 Mason's Avenue**

**Croydon CR9 3NB (GB)**

(54) **Reactive coalescents**

(57) Compounds useful as reactive coalescents and methods of improving the stability of reactive coales-

cents are disclosed. A process for the preparation of low molecular weight polymers which are also useful as reactive coalescents is also disclosed.

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## Description

This invention relates to compounds which are useful as reactive coalescents, methods of improving the stability of reactive coalescents, and a process of preparing low molecular weight polymers which are useful as reactive coalescents.

Water based polymers having a low glass transition temperature ("Tg") can be formulated into coatings without plasticizers. These coatings often have inadequate hardness for many applications. Many applications, such as gloss and semigloss paint formulations, require the properties of a hard polymer, i.e. a polymer with a Tg significantly above the ambient temperature. To meet these needs, a volatile coalescent or plasticizer is typically used to achieve film formation. The use of these volatile solvents in coatings is coming under scrutiny due to pollution and odor problems.

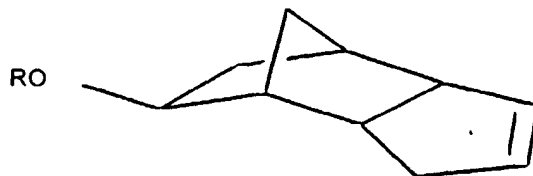
Attempts have been made to use "reactive coalescents". Reactive coalescents are compounds which aid in film formation similar to the conventional coalescent but are non-volatile and react to become part of the final coating.

U.S. Pat. No. 4,141,868 discloses the use of dicyclopentenylloxyethyl methacrylate ("DCPOEMA") as a reactive coalescent. Vinyl reactive coalescents are reactive coalescents which contain a vinyl group. DCPOEMA, dicyclopentenylloxyethyl acrylate ("DCPOEA"), dicyclopentenylloxy acrylate ("DCPOA"), and dicyclopentenylloxy methacrylate ("DCPOMA") are examples of vinyl reactive coalescents.

Despite the disclosure, there is a continuing need for stable reactive coalescents for use in coating compositions.

We have surprisingly found that stable reactive coalescents for use in coating compositions can be obtained by altering the structure of the reactive coalescent or adding an inhibitor to the reactive coalescent.

In a first aspect, the present invention provides compounds of formula I:



(I)

Wherein:

$R = -(CH_2)_nR^1$ ,  $-(CH_2)_2O(CH_2)_2OH$ ,  $-(CH_2CH_2O)_2(CH_2)_3OH$ ,  $-CH_2CH_2OCOR^2$ ,  $-(CH_2)_2O(CH_2)_2O(CH_2)_3CH_3$ ,  $-CH_2CH(CH_3)-OH$ ,  $-CH_2CH_2OCO(CH_2)_4COOR^3$ , dicyclopentenylloxyethane;

$R^1$  is selected from H, OH and  $CH_3$ ;

$R^2 = (C_1 - C_6)$  straight chain or branched alkyl;

$R^3$  is selected from  $CH_3$  and dicyclopentenylloxyethane;

and  $n = 2$  to 10.

In a second aspect, the present invention provides a process for the preparation of polymers comprising:

providing a compound selected from the group consisting of DCPOEA, DCPOEMA, DCPOA, and DCPOMA;

providing a solvent selected from the group consisting of water, acetone, methanol, isopropanol, propionic acid, acetic acid, toluene, hexane, ethyl acetate, methylethyl ketone, dimethyl formamide, dimethylsulfoxide, and combinations thereof,

providing an initiator;

forming a reaction mixture by admixing the compound selected from the group consisting of DCPOEA, DCPOEMA, DCPOA, and DCPOMA, the solvent, and the initiator; and

passing the reaction mixture through a heated zone wherein the reaction mixture is maintained at a temperature of at least 175°C for from 0.1 seconds to 300 seconds to form a liquid polymer with a number average molecular weight of from 450 to 10,000.

In a third aspect, the present invention provides a method of use comprising:

providing an emulsion polymer;

providing a liquid polymer prepared from a compound selected from the group consisting of DCPOEA, DCPOEMA,

DCPOA, and DCPOMA, the liquid polymer having a number average molecular weight of from 450 to 10,000; forming a coating composition by admixing the liquid polymer having a number average molecular weight of from 450 to 10,000 with the emulsion polymer; and; applying said coating composition to a substrate wherein the coating composition forms a continuous film on the substrate.

In another embodiment, the present invention provides a composition comprising:

from 1% to 25% of a vinyl reactive coalescent based on a weight reactive coalescent to total weight of composition basis;

from 1 ppm to 10,000 ppm of an inhibitor selected from the group consisting of 4-methoxyphenol, 2,2,6,6-tetramethyl-1-piperidinyloxy, 4-oxo-2,2,6,6-tetramethyl-1-piperidinyloxy, and di-tertiary butyl nitroxyl, based on a weight inhibitor to total weight of composition basis; and

from 74% to 99% of an emulsion polymer based on a weight emulsion polymer to total weight of composition basis.

In another embodiment, the present invention provides a method for improving the stability of a vinyl reactive coalescent comprising:

providing an emulsion polymer;

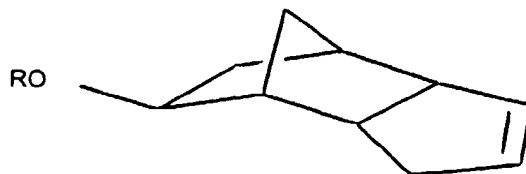
passing the emulsion polymer through a diafiltration apparatus; and

charging the emulsion polymer which has been passed through the diafiltration apparatus with a vinyl reactive coalescent.

In another embodiment, the present invention provides a coating composition comprising:

an emulsion polymer; and

a compound selected from the compounds of Formula I



( I )

Wherein:

R =  $-(CH_2)_nR^1$ ,  $-(CH_2)_2O(CH_2)_2OH$ ,  $-(CH_2CH_2O)_2(CH_2)_3OH$ ,  $-CH_2CH_2OCOR^2$ ,  $-(CH_2)_2O(CH_2)_2O(CH_2)_3CH_3$ ,  $-CH_2CH(CH_3)-OH$ ,  $-CH_2CH_2OCO(CH_2)_4COOR^3$ , dicyclopentenylloxyethane;

$R^1$  is selected from H, OH and  $CH_3$ ;

$R^2$  =  $(C_1 - C_6)$  straight chain or branched alkyl;

$R^3$  is selected from  $CH_3$  and dicyclopentenylloxyethane; and  $n = 2$  to 10,

and a liquid polymer prepared from a compound selected from the group consisting of DCPOEA, DCPOEMA, DCPOA, and DCPOMA, the liquid polymer having a number average molecular weight of from 450 to 10,000.

The inhibitors useful in this invention include hydroquinone ("HQ"), 4-methoxyphenol ("MEHQ"), phenothiazine ("PTZ"), 2,2,6,6-tetramethyl-1-piperidinyloxy ("4-HT"), and 4-oxo-2,2,6,6-tetramethyl-1-piperidinyloxy, are available from Aldrich Chemical Company. Di-tertiary butyl nitroxyl is available from Nova Molecular Technologies, Lake Geneva, Wisconsin. The inhibitors are typically used at levels of from 1 ppm to 10,000 ppm on a weight inhibitor to total weight of the composition basis. Preferred are inhibitor levels of from 100 ppm to 1,000 ppm. More preferred are inhibitor levels of from 200 ppm to 700 ppm.

The reactive coalescents of this invention are prepared by processes known in the art. For example, direct esterification, transesterification or the use of acid halides may be employed to convert alcohols to esters of this invention. Examples of compounds of Formula I include, but are not limited to: dicyclopentenylloxyethyl acetate, dicyclopentenylloxyethyl butyrate, dicyclopentenylloxy-2-(2-hydroxyethoxy)ethane, dicyclopentenylloxy-2-(2-butoxyethoxy)ethane, di-

cyclopentenylhexanol, dicyclopentenylhexane, dicyclopentenylbutane, dicyclopentenyl (2-methyl)propane, and 1,2-bis (dicyclopentenyl)ethane.

The low molecular weight polymers of this invention may be useful as reactive coalescents, and may be prepared by conventional techniques, such as solution or emulsion polymerization, which are well known in the art. The low molecular weight polymers may also be prepared by the high temperature, low residence time process of this invention. In the process of this invention, a reaction mixture is formed by combining at least one compound selected from DCPOEA, DCPOEMA, DCPOA, and DCPOMA and optionally, compatible monomers with a solvent and an initiator. Suitable solvents include, but are not limited to water, acetone, methanol, isopropanol, propionic acid, acetic acid, toluene, alkanes such as hexane; esters such as ethyl acetate; methylethyl ketone, dimethyl formamide, dimethylsulfoxide, and combinations thereof. The process of this invention may be run in the absence of solvent.

Initiators are compounds which initiate the polymerization of monomers. Suitable initiators for the present invention are any conventional initiators. Among the suitable initiators that may be used are thermal free-radical initiators, such as hydrogen peroxide, certain alkyl hydroperoxides, dialkyl peroxides, persulfates, peresters, percarbonates, ketone peroxides and azo initiators. Specific free-radical initiators include, for example, hydrogen peroxide, tert-butyl hydroperoxide, di-tert-butyl peroxide, ammonium persulfate, potassium persulfate, sodium persulfate, tert-amyl hydroperoxide and methyl ethyl ketone peroxide. The free-radical initiators are typically used in amounts of 0.5 to 25% based on the total monomer weight.

Redox initiator systems may also be used. Redox initiator systems include reducing agents, for example, sodium bisulfite, sodium sulfite, hypophosphites, phosphites, isoascorbic acid, sodium formaldehyde-sulfoxylate and hydroxylamines, used in conjunction with suitable oxidizing agents, such as the thermal free-radical initiators noted above. The redox initiator systems are typically used in amounts from 0.05 to 10%, preferably from 0.5 to 5%, based on the weight of total monomer. Combinations of initiators may be used.

The process of this invention is typically run at a temperature of from 175°C to 500°C. Preferred is a temperature of from 225°C to 450°C. It is preferred to conduct the polymerization at a pressure of from 1,000 to 5,000 pounds per square inch ("psi"), and more preferably at from 3,200 to 4,200 psi. By low molecular weight polymers is meant polymers typically with number average molecular weights from 450 to 10,000. Preferred are polymers with number average molecular weights from 450 to 5,000. More preferred are polymers with number average molecular weights from 450 to 3,000.

Compatible monomers are monomers which are miscible with DCPOEA, DCPOEMA, DCPOA or DCPOMA. Compatible monomers include, but are not limited to: methyl acrylate, ethyl acrylate, butyl acrylate, t-butylaminoethyl methacrylate, dimethylaminoethyl methacrylate, isobornyl methacrylate, styrene, vinyl toluene, methacrylic acid, methyl methacrylate, butyl methacrylate, isobutyl methacrylate, allyl methacrylate, lauryl methacrylate, and 2-ethylhexyl methacrylate.

The reactive coalescents are typically used at levels of from 1% to 25% on a weight reactive coalescent to total weight of composition basis. Preferred are reactive coalescent levels of from 2% to 20%. More preferred are reactive coalescent levels of from 5% to 15%.

This invention may be applied to any emulsion polymer. The preparation of emulsion polymers is known in the art, see for example U.S. Pat. No. 5,346,954.

The following is a list of products used within this invention and their sources:

Product	Source
Tamol® 165	Rohm and Haas Company
Triton® CF-10	Union Carbide
Drew® ZV-22	Ashland Chemical Company
RM-2020NPR	Rohm and Haas Company
Ti-Pure® R-706	DuPont Chemical Company
Rhoplex® EXP-3361	Rohm and Haas Company
Scotch® Tape	Minnesota Mining and Manufacturing Company
Tamol® 731	Rohm and Haas Company
BYK-22	BYK-Chemie GmbH
GR-7M	Union Carbide
Triton® XN-45S	Union Carbide

The following examples are intended to demonstrate the compounds and compositions of this invention and their use as stable reactive coalescents.

Example 1 - Improved Stability Of DCPOEMA

Reactive coalescents containing unsaturated vinyl groups can polymerize in the can due to residual impurities in the polymer. The poor stability of the coalescent manifests itself as loss of film forming properties of the formulated coating over time. DCPOEMA is a reactive coalescent which contains 100 ppm hydroquinone as purchased. Various inhibitor types and levels were added to DCPOEMA prior to use in a coating composition. The reactive coalescent and inhibitor mixtures were prepared prior to the compositions by adding DCPOEMA to a fixed amount of solid grade inhibitor. The mixtures were then post-added to a portion of a master mix of paint 1. A portion of each sample was stored at 140°F for 10 days. The minimum film forming temperature ("MFFT") was measured for each sample. If the DCPOEMA was stable, the MFFT remained low when compared to the control sample. If the DCPOEMA was unstable, the MFFT was elevated when compared to the control. The DCPOEMA was considered to be stable if the MFFT increased less than 5°C after the sample was stored at 140°F for 10 days. The results of the tests are shown in Table 1.

Paint 1

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A master paint formulation containing no coalescent was prepared as follows:

18 g water, 1.61 g Tamol® 165, 0.50 g Triton®CF-10, 0.25 g Drew®ZV-22,

20

3.75 g RM-2020NPR, and 45.0 g T-Pure®R-706 were ground with a high speed disperser for 15 minutes. To the mixture was added 131.27 g EXF-3361,

0.25 g Drew®ZV-22, 1.18 g RM2020NPR, and 40.25 g water with further mixing.

25

Sample 1 was a control sample of Paint 1 with DCPOEMA, but no additional inhibitor.

Sample 2 was prepared by combining 7.55 g of a mixture of 0.29 g of solid grade 4-HT dissolved in 57.71 g of DCPOEMA with 244.06 g of paint 1.

Sample 3 was prepared by combining 7.55 g of a mixture of 0.60 g of solid grade 4-HT dissolved in 59.40 g of DCPOEMA with 244.06 g of paint 1.

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Sample 4 was prepared by combining 7.55 g of a mixture of 1.20 g of solid grade 4-HT dissolved in 58.80 g of DCPOEMA with 244.06 g of paint 1.

Sample 5 was prepared by combining 7.55 g of a mixture of 2.37 g of solid grade 4-HT dissolved in 56.88 g of DCPOEMA with 244.06 g of paint 1.

35

Sample 6 was prepared by combining 7.55 g of a mixture of 0.30 g of MEHQ dissolved in 59.70 g of DCPOEMA with 244.06 g of paint 1.

Sample 7 was prepared by combining 7.55 g of a mixture of 0.60 g of MEHQ dissolved in 59.40 g of DCPOEMA with 244.06 g of paint 1.

Sample 8 was prepared by combining 7.55 g of a mixture of 1.16 g of MEHQ dissolved in 56.84 g of DCPOEMA with 244.06 g of paint 1.

40

Sample 9 was prepared by combining 7.55 g of a mixture of 2.42 g of MEHQ dissolved in 58.08 g of DCPOEMA with 244.06 g of paint 1.

Sample 10 was prepared by combining 7.55 g of a mixture of 0.24 g of HQ dissolved in 57.71 g of DCPOEMA with 244.06 g of paint 1.

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Sample 11 was prepared by combining 7.55 g of a mixture of 0.60 g of HQ dissolved in 59.40 g of DCPOEMA with 244.06 g of paint 1.

Sample 12 was prepared by combining 7.55 g of a mixture of 1.19 g of HQ dissolved in 58.31 g of DCPOEMA with 244.06 g of paint 1.

Sample 13 was prepared by combining 7.55 g of a mixture of 0.31 g of PTZ dissolved in 61.69 g of DCPOEMA with 244.06 g of paint 1.

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Sample 14 was prepared by combining 7.55 g of a mixture of 0.61 g of PTZ dissolved in 60.39 g of DCPOEMA with 244.06 g of paint 1.

Sample 15 was prepared by combining 7.55 g of a mixture of 1.21 g of PTZ dissolved in 59.29 g of DCPOEMA with 244.06 g of paint 1.

55

The MFFT test was performed as follows:

Scotch®Tape biaxially oriented polypropylene was placed on an ICI Sheen MFFT Bar SS-3300. Samples were drawn down over the tape using a 1 inch cube Sheen Film Applicator with a gap size of 75 micron. After 60 to 90

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minutes, the MFFT was read as the point where the film becomes continuous and not cracked.

The Low Temperature Film Formation test was performed as follows:

5 Samples were painted by brush onto white pine boards. The paint was applied in strips perpendicular to the length of the board. Each sample was weighed to provide a spread rate of 41.8 m<sup>2</sup>/liter. The painted boards were dried for 24 hours at 40°F / 70% relative humidity. A magnifying glass was used to determine the degree of cracking in the paint film. The degree of cracking was reported according to the following scale:

10 = none, 9 = trace, 8 = trace/slight, 7 = slight, 6 = slight/moderate, 5 = moderate, 4 = moderate/heavy, 3 = heavy, 2 = heavy/very heavy, 1 = very heavy.

A score of 10 is considered to be passing.

Table 1

Sample	Inhibitor	Inhibitor Level	MFFT (°C)	
			Initial	10 Days@140F
1	None	None	5.1	>18
2	4-HT	625 ppm	5.1	5.6
3	4-HT	1250 ppm	5.3	5.6
4	4-HT	2500 ppm	5.3	5.6
5	4-HT	5000 ppm	5.3	5.6
6	MEHQ	625 ppm	4.9	5.6
7	MEHQ	1250 ppm	4.9	6.4
8	MEHQ	2500 ppm	4.9	7.6
9	MEHQ	5000 ppm	4.5	7.6
10	HQ	625 ppm	7.8	>18
11	HQ	1250 ppm	8.0	>18
12	HQ	2500 ppm	9.2	>18
13	PTZ	625 ppm	8.0	>18
14	PTZ	1250 ppm	7.8	>18
15	PTZ	2500 ppm	9.2	>18

35 The results demonstrate that 4-HT and MEHQ improve the stability of DCPOEMA. HQ and PTZ did not improve the stability of DCPOEMA.

### Example 2 - The Level Of Inhibitor Required To Stabilize DCPOEMA

40 The level of 4-HT required to provide stability in commercial polymers was evaluated. Samples were prepared by adding 4-HT to latex, this was called a modified latex. The modified latex was then formulated into paints. Sub-samples of Samples 16 - 19 were stored at 140°F for 30 days. Sub-samples of Samples 20 - 23 were stored at 140°F for 10 days. The MFFT was measured for each Sample. The results are shown in Tables 2 and 3.

#### Paint 2

Paint 2 was prepared for Samples 16 - 19 as follows:

72 g water, 12 g Tamol®731, 2 g Triton®CF-10, 2 g BYK 22,  
50 20 g RM-2020NPR, and 300 g Ti-Pure®R-706 were ground with a high speed disperser for 15 minutes. To the mixture was added 106.32 g water, 2 g GR-7M, 544.14 g modified Rhoplex®AC-261, 26.84 g DCPOEMA, 2 g BYK-22, and 10 g water with further mixing.

#### Paint 3

55 Paint 3 was prepared for Samples 20 - 23 as follows:

72 g water, 12 g Tamol®731, 2 g Triton®CF-10, 2 g BYK 22,

and a Brookfield viscosity (LVT viscometer, 30 rpm, 25 °C) of 60 cps.

Table 4

	ME #1	ME #2
DI water	121.3 g	260.0 g
Triton®XN-45S	15.8 g	25.4 g
Butyl Acrylate	326.7 g	381.2 g
Methyl Methacrylate	388.5 g	517.3 g
Allyl Methacrylate	7.3 g	
Methacrylic Acid	3.6 g	27.2 g
Acetoacetoxy		163.4 g
Methacrylate		

The microfiltration unit was an Industrial Maximate manufactured by Filtron. The main feed tank was a 5 gallon polypropylene tank. The recirculating pump was an M-1 diaphragm pump manufactured by Wildon. An in-line water filter was positioned after the pump. The microfiltration membrane was an Omega series modified polyether sulfone. The molecular weight cut-off for the membrane was about 30,000. The channel size of the membrane was 40 mil. The solution being filtered was recirculated from the main feed tank to the microfiltration membrane and back to the main feed tank by the pump. A 2.5 gallon polypropylene carboy was used to collect the permeate as it was produced on the other side of the microfiltration membrane. A feed pump manufactured by Fluid Metering Pump Inc. was used to add deionized water to the main feed tank to maintain the fluid volume in the tank during filtration.

A solution was prepared by mixing 3,500 g of JH-2479 with 3,500 grams of deionized water. This solution was recirculated across the microfiltration membrane using the equipment described above. During the filtration process 10,500 g of deionized water was added to the main feed tank at a rate that equaled the rate of permeate that was collected on the other side of the microfiltration membrane. After the 10,500 g of deionized water was added and 10,500 g of permeate was collected, the solution was allowed to recirculate in the microfiltration unit until another 3,685 g of permeate was collected. The sample was drained from the unit.

The final product had a solids content of 44.2%, particle size by Brookhaven BI-90 (light scattering) of 194 nm., and a Brookfield viscosity (LVT viscometer, 30 rpm, 25 °C) of 68 cps.

Sample 24 was a control paint sample of JH2479 charged with DCPOEMA. Sample 25 was the diafiltered paint sample of JH2479 charged with DCPOEMA. Samples were stored at 140°F for 10 days. The concentration of DCPOEMA was measured by Gas Liquid Chromatography at 0, 5, and 10 days storage. For this test, the reactive coalescent was considered stable if greater than 50% of the reactive coalescent remained after 5 and 10 days storage at 140°F. Preferred is greater than 60% of the reactive coalescent remaining after 5 and 10 days storage at 140°F. Most preferred is greater than 75% of the reactive coalescent remaining after 5 and 10 days storage at 140°F. The results are shown in Table 5.

Table 5

Concentration of DCPOEMA in Coating Compositions					
Sample	Polymer	Initial	2 Week RT	5day@140F	10day@140F
24	JH2479	29,030 ppm	28,200 ppm	2,600 ppm	1,365 ppm
25	Diafiltered	28,875 ppm	27,863 ppm	24,982 ppm	21,521 ppm

The results demonstrate that diafiltration of the polymer prior to formulation with DCPOEMA improves the stability of DCPOEMA in the wet coating composition.

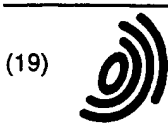
#### Example 4 - Analogs Of DCPOEMA

##### Sample 26 - Dicyclopentenylxyethyl Butyrate

A solution of 155.2 grams (0.8 moles) of dicyclopentenylxyethanol and 201.5 grams (1.96 moles) of methyl butyrate was charged to a 500 ml, 3-necked flask equipped with a thermometer, a mechanical stirrer, and a 1 inch diameter - 10 plate Oldershaw column fitted with a distillation head, distillate rate removal - vapor temperature controller and a graduated distillate receiver. The solution was stirred and heated to reflux (108 - 112 °C / 700 mm of Hg). The water - methyl butyrate azeotrope ( 18.3 g) was distilled from the reaction mixture. At the conclusion of the dehydration, the







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Ambler, PA 19002 (US)
- **Bowe, Michael Damian**  
Newtown, PA 18940 (US)
- **Weir, William David**  
Levittown, PA 19056 (US)
- **Wolfsberger, Martha Alice Harbaugh**  
Perkasie, PA 18944-4127 (US)

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(71) Applicant: **ROHM AND HAAS COMPANY**  
Philadelphia, Pennsylvania 19106-2399 (US)

(74) Representative: **Buckley, Guy Julian et al**  
**ROHM AND HAAS (UK) LTD.**  
European Operations Patent Department  
Lennig House  
2 Mason's Avenue  
Croydon CR9 3NB (GB)

(72) Inventors:  
• **Speece, David Gerald, Jr.**  
Reading, PA 19606 (US)

(54) **Reactive coalescents**

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DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (In Cl. 8)
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6 X	DE 12 78 431 B (CONSORTIUM FÜR ELEKTROCH. IND.) * example 3 * ---	1	
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The present search report has been drawn up for all claims			
Place of search MUNICH		Date of completion of the search 18 November 1998	Examiner Goetz, G
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The present search report has been drawn up for all claims			
Place of search MUNICH		Date of completion of the search 18 November 1998	Examiner Goetz, G
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons &amp; : member of the same patent family, corresponding document</p>			

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